

Certificate of Analysis

Standard Reference Material® 1616b

Sulfur in Kerosene (Low Level)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of instruments used in the determination of total sulfur in fuel oils or materials of a similar matrix. SRM 1616b consists of a special low sulfur kerosene (No. 1-K) suitable for use in non flue-connected burner appliances and for use in wick-fed illuminating lamps, as described in ASTM D 3699-92 Specification for Kerosene [1]. A unit of SRM 1616b consists of 100 mL of the low sulfur kerosene in an amber glass bottle.

Certified Value: The certified sulfur content, provided in Table 1, is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [3]. Homogeneity testing was performed using X-ray fluorescence spectrometry (XRF). The expanded uncertainty for the certified value for sulfur is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [4]. The coverage factor, k = 2.23, corresponds to a t factor obtained from the t-distribution for approximately 10.3 degrees of freedom.

Table 1. Certified Value (mass fraction)

Sulfur: $8.41 \text{ mg/kg} \pm 0.12 \text{ mg/kg}$

Information Values: Information values are provided in Table 2 for additional properties of SRM 1616b. The values are not certified and are given to provide additional information on the matrix, but insufficient information is available to assess adequately the uncertainties associated with the values [3].

Expiration of Certification: The certification of this SRM is valid until **31 December 2015**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in the certificate, see ("Instructions for Use"). However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was provided by W.R. Kelly and G.C. Turk of the NIST Analytical Chemistry Division.

Analytical measurements by ID-TIMS for certification were performed by W.R. Kelly, J.L. Mann, and R.D. Vocke and homogeneity testing by X-ray fluorescence spectrometry was performed by A.F. Marlow and J.R. Sieber of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief Analytical Chemistry Division

Robert L. Watters, Jr., Chief Measurement Services Division

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Statistical consultation for this SRM was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

Bottling was performed under the supervision of M.P. Cronise of the NIST Measurement Services Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The kerosene for this SRM was donated by BP Products North America, Naperville, IL.1

INSTRUCTIONS FOR USE

Each SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 150 mg should be used. The tightly capped bottle should be stored under normal laboratory conditions away from direct sunlight.

Table 2. Information Values for Selected Properties

Physical Property Test ^(a)	ASTM Standard Used	Result
Density @ 15 °C @ 60 °F	D 1250-80 (1990) ^{€1} D 4052-96	827.1 kg/m ³ 39.5 API
Flash Point	D 93 (A)-94	58.9 °C
Kinematic Viscosity @ 40 °C	D 445-94 ^{€1}	$1.617 \times 10^{-6} \text{ m}^2/\text{s} (1.617 \text{ cSt})$
Carbon	D 5291-92	84.0 %
Hydrogen	D 5291-92	13.1 %

⁽a) These properties were determined by a commercial firm under contract to NIST using ASTM methods. The results are **NOT** certified and are provided as additional information on the matrix.

ASTM Standards

D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester	
D 4052-96	Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter	
D 445-94 ^{€1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids	
(the Calculation of Dynamic Viscosity)		
D 1250-80 $(1990)^{\epsilon_1}$	Standard Guide for Petroleum Measurement Tables	
D 2274-94	Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)	
D 5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen	
in Petroleum Products and Lubricants		

 $^{epsilon^{-1}}$: A superscript epsilon (E) indicates an editorial change since the last revision or reapproval. The superscript number refers to the specific change.

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¹ Certain commercial organizations, services, equipment, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the organizations, services, materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] ASTM D 975-97; Standard Specification for Diesel Fuel Oils; Annual Book of ASTM Standards, Vol. 05.01, West Conshohocken, PA (1998).
- [2] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D., Jr.; Chen, L.-T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem., Vol. 66, pp. 2505–2513 (1994).
- [3] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Washington, DC, p. 16 (2000).
- [4] ISO; Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, lst ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Certificate Revision History: 12 September 2006 (Editorial changes); 10 March 2005 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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